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Vitrinite-Reflectance Procedures Followed by M.J. Pawlewicz

Sample-preparation procedures depend on the lithology and organic-carbon content of the sample. Coals require no special preparation other than crushing to ~0.1 mm and casting in epoxy. Shales and siltstones similarly require no special processing, but their kerogen must be concentrated by the crush-and-float technique described below. Sandstones and carbonates require acid digestion before kerogen concentration. Sandstones are digested successively in hydrochloric and hydrofluoric acids; carbonate samples generally require only a hydrochloric-acid treatment. For shales, about 20-40 g of material are required, depending on organic content. Sandstones and siltstones require about 40 g of material, and carbonate samples should consist of 60-70 g of material.

The crush-and-float technique (see Barker, 1982; Barker and Pawlewicz, 1986b) begins with crushing of shales and siltstones or acid digestion of sandstones and carbonates. Before milling in a micropulverizer, large (>10 cm) pieces of rock are reduced to 2–4 cm in diameter by hand, then crushed by a jaw-crusher to yield a uniform particle size of 2-5 mm. The sample is then milled in a micropulverizer, yielding particles ~150 µm in diameter (100 mesh). The crushed sample (shales and siltstones) or residue after acid digestion (sandstones and carbonates) is then placed into a 50-mL polycarbonate test tube for kerogen concentration. Enough $ZnBr_2$ (specific gravity 2.1 g/cm^3) is added to each tube to just wet the sample. Each tube in turn is then stirred with a stirring motor and paddle turning at about 700 RPM. Additional $ZnBr_2$ is then added to yield a thin slurry, and the volume of material in each tube is kept approximately equal for all samples to facilitate balancing the tubes before centrifugation.

The $ZnBr_2$-sample slurries are centrifuged for 12 minutes at about 2,800 RPM, which separates organic material (which floats in $ZnBr_2$) from inorganic material (which settles to the bottom of the tube). The supernatant is decanted, saved, and labeled; the inorganic material is discarded. The supernatant is then returned to the centrifuge tubes, diluted with water, and centrifuged for 5 minutes to wash the sample of $ZnBr_2$. The supernatant is discarded, and the washing is repeated two more times.

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The washed organic material is then set on a hot plate at ~28°C to dry overnight. The kerogen concentrate (or crushed coal) is then cast in epoxy on standard petrographic slides, using the method described by Baskin (1979). A surface parallel to the slide is cut in the epoxy by use of a thin-section machine. The saw marks caused by this procedure are removed by using a 600-grit sandpaper and water. Slides are polished in two steps (see Pawlewicz, 1987). The first step is a 90-second treatment, using 0.5-µm grit (alpha alumina) on a nap-free cloth with the lap turning at 60 RPM. The slide is removed and thoroughly cleaned of all polishing compound, then polished for an additional minute, using CeO_2 on a napped cloth also revolving at 60 RPM. After rinsing with water, the slides are cleaned in an ultrasonic cleaner and blown dry with a stream of air. The slides are then placed in a desiccator until ready for use.

Several assumptions are made when making vitrinite reflectance measurements. The first assumption is that the organic matter in the sample is representative of that incorporated into the sediment during deposition. Separating material recycled from older rock units from indigenous material (derived from vegetation coeval with sediment deposition) is an important consideration. Availability of the organic material and factors controlling local sedimentation determine the distribution of organic material in the depositional environment, as well as the type of organic material preserved. The net result is that many distinct populations of organic material may be found in any lithologic unit. Homogenous vitrinite populations do occur, but infrequently. The second assumption is that organic material is distributed evenly throughout the slide and that measurements taken at any point on the polished section will yield the same mean value (to within ~5 percent). The presence of recycled material, or material other than terrestrial organic matter, is noted but not quantified during analysis. In samples in which organic material is sparse it is even more difficult to select the indigenous population while rejecting recycled or nonindigenous material.

Vitrinite-reflectance determinations were made on a Zeiss Universal microscope, fitted with an

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MPC-65 microscope controller and interfaced with an IBM OS-2 Model 60 computer running the Zeiss reflectance program PHOTAN. This program allows for the immediate compilation of reflectance data, storage on disk in several formats, and printing of the data and histogram. Our usual procedure is to perform one scan per sample, measuring the reflectance of all the pertinent material as defined above. The mean of the individual values is usually taken as representing the thermal maturity of the sample, but rare values that are significantly different from the modal population may be discounted. Extensive editing of data or repeat analyses of any sample are not allowed.

Bimodal samples, where there is a distinct break in the reflectance values in the histograms, present special problems in interpretation. If the sample is from outcrop, or core from a borehole, the lowest reflectance values from terrestrial organic material must be the correct thermal-maturity indicator. Higher values are probably recycled material. When the separation of reflectance values is vague or the values range widely, then the decision usually is to use the mean of all readings. Operator experience obviously is beneficial in the evaluation of complex vitrinite-reflectance populations.

Conodont Color-Alteration-Index Procedures Followed by A.G. Harris

Detailed descriptions of techniques for concentrating conodonts from rock, sediment, and residue matrix were given by Collinson (1963), Stone (1987), and Harris and Sweet (1989). The sample-preparation techniques used in this study, with some exceptions, followed those of Harris and Sweet (1989). Most of the samples reported here are from carbonate rocks. Samples from outcrops usually consisted of 3-8 kg of material, but well samples (generally cuttings) were much smaller (0.1–1 kg). Samples were crushed to fragments ~2 cm in diameter in a standard rock crusher that was scrubbed with a wire brush and thoroughly cleaned with compressed air between samples. Well cuttings did not require crushing but were wet-sieved and cleaned of contaminants (including metal shavings, which were removed with a hand-held magnet). Crushed samples or well cuttings were placed in plastic pans and covered with 6–7% glacial acetic acid (limestones) or 4–5% formic

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acid (dolostones). After 1–3 days, or when all CO_2 generation ceased, the insoluble reside was washed through nested 20- and 200-mesh sieves. The >20-mesh fraction was reacidifed and washed repeatedly until <100 g of >20-mesh rock remained. Samples containing abundant noncarbonate minerals were crushed to particles <1 cm diameter before re-acidification. The 20-200 mesh (75–850 µm) fraction was oven-dried at <50°C. Conodonts were concentrated from chert, following the general recommendations in Orchard (1987). Samples weighing 20–200 g were crushed to <2-cm diameter and placed in a 1-L polypropylene beaker in a fume hood and immersed on a 4–5 % hydrofluoric acid solution. After 24 hours, sodium carbonate was added to the solution to neutralize the remaining acid. The insoluble residue was then washed gently through stainless-steel sieves and dried as above.

Because conodonts generally have a specific gravity of 2.87 to 3.0 g/cm^3, heavy-liquid separation techniques were used to concentrate them from the 75–850-µm acid-insoluble residue. In a fume hood, the residue was poured into a transparent, 2-L separatory funnel containing tetrabromoethane adjusted to a density of 2.865 g/cm^3 for limestone samples or 2.88 g/cm^3 for dolostones, and stirred at 15-minute to 1-hour intervals about 5–10 times. The heavy-mineral concentrate was drawn off onto filter papers, thoroughly washed with acetone, and air-dried in a hood. If concentrates contained >20% iron-bearing minerals (exclusive of unweathered pyrite) they were subjected to magnetic separation, using a Frantz-type electromagnetic separator and the procedures outlined by Dow (1965) and Stone (1987), to separate phosphatic minerals from nonphosphatic, predominantly iron-bearing minerals. If the heavy-mineral concentrate contained significant amounts of nonmagnetic minerals (pyrite, barite, fluorite, etc.), additional heavy-liquid separation was performed, using methylene iodide adjusted to a specific gravity of 3.1 g/cm^3, in which the relatively less dense conodonts float.

The final concentrate was then picked for conodonts by sprinkling the residue onto a picking tray with a numbered grid and white background to form a loose layer one grain thick. The conodonts

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were picked under a binocular zoom microscope at 20–50x magnification, using a fine wetted brush. The conodonts were transferred to a microfossil slide very lightly coated with water-soluble glue. Conodont color-alteration-index (CAI) values were determined, following the procedures given in Epstein and others (1977) and Rejebian and others (1987). A set of CAI standards was used for indexing: each CAI value is represented by 10–30 naturally and experimentally altered, morphologically diverse specimens. The standard used for this study includes clusters of conodonts with CAI values of 1, 1.5, 2, 3, 4, 4.5, 5, 5.5, 6, 6.5, 7, 7.5, and 8. The set of CAI standards, mounted on transparent glass set on white paper, was placed under a binocular zoom microscope. The field was illuminated with white light from a Tiyoda microscope light, using an 8V, 5A bulb. Relatively thin, unornamented specimens containing as little white matter as possible were selected for indexing. These specimens were placed next to the CAI standards under the microscope, and a best match was determined. When possible, at least five specimens representing different morphotypes were indexed for CAI values of 1 through 5. Some samples contained conodonts representing two CAI values (e.g., 4 and 4.5, indexed as 4–4.5) whereas some others, commonly from hydrothermal or contact-metamorphic regimes, yielded conodonts having a discontinuous range of CAI values (e.g., 2, 3, and 6). The latter were indexed as having the full range of values (e.g., 2–6). For samples containing conodonts with CAI values above 5, similar procedures were followed, except when a continuous range of CAI values was present (e.g., 5.5, 6, 6.5, and 7). For these samples, every specimen was indexed, and the sample was assigned the mean CAI value.

Some conodonts extracted from chert using hydrofluoric acid could not be used for CAI analysis because of chemical alteration. This sample-preparation technique produces conodonts with a wide range of preservation quality so that a sample may yield some specimens that are brittle, corroded, and bleached and others that are unaffected to only slightly affected by the acid treatment; some conodonts must dissolve completely. The unaffected conodonts probably have had the shortest exposure to the hydrofluoric acid. Only chemically unaltered specimens were used for CAI analysis.

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Conodonts exposed on surfaces of indurated siliciclastic rocks could not be freed from their matrix. Nevertheless, these samples were indexed under the microscope by placing morphologically similar CAI standards on the rock next to the exposed specimens and determining a best match. Such determinations are less accurate than those made on matrix-free specimens, particularly for CAI values less than 5.

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